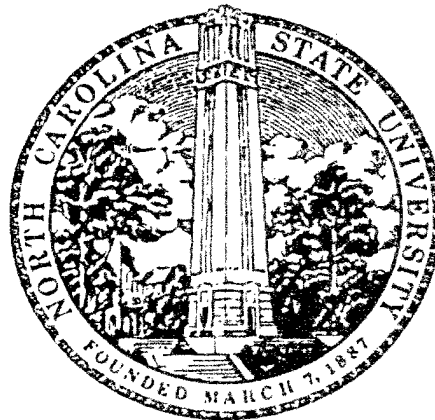
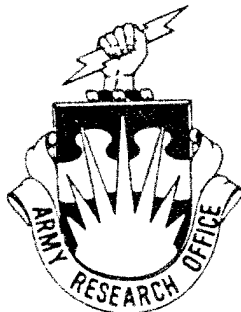


Final Report
on
ARO-Sponsored Workshop
**ENHANCED SYNTHESIS, PROPERTIES AND
PROCESSING OF MATERIALS WITH ELECTRIC
AND MAGNETIC FIELDS**

Sponsored by
U. S. Army Research Office
ARO Proposal No. 36285-MS-CF
Grant No. DAAG55-98-1-0484

Saint Christopher Conference
Johns Island, South Carolina
May 16 - 19, 1999



Hans Conrad, Yusef Fahmy and Di Yang
Materials Science and Engineering Department
North Carolina State University
Raleigh, NC 27695-7907

Wilbur Simmons
Army Research Office
Research Triangle Park, NC 27709-2211

September 15, 1999

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On
ARO-Sponsored Workshop
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Final Report

On
ARO-Sponsored Workshop (Proposal No. 36285-MS-CF;
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**ENHANCED SYNTHESIS, PROPERTIES AND
PROCESSING OF MATERIALS WITH ELECTRIC
AND MAGNETIC FIELDS**

Hans Conrad, Yusef Fahmy and Di Yang
Materials Science and Engineering Department
North Carolina State University
Raleigh, NC 27695-7907

Wilbur Simmons
Army Research Office
Research Triangle Park, NC 27709-2211

Abstract

The program, participants (34 from seven countries) and abstracts of the papers presented at the workshop are given. The papers cover a wide range of phenomena in which electric and magnetic fields exert a significant influence and which offer potential for beneficial technological applications. Comments and recommendations by the participants are presented. These include an expression of the dire need to better understand the mechanisms involved and the use of electric and magnetic fields to: (a) produce new materials, (b) process amorphous, non-equilibrium, nanocrystalline and composite materials, and (c) fabricate special architected structures. Also, the establishment of a Center on the general subject of the effects of electric and magnetic fields was strongly urged to promote interaction and cooperation among the research workers in the field and to develop the state-of-the-art required for technological applications.

1. Introduction

The external parameters generally considered in the synthesis, processing and properties of materials are the temperature, pressure (or stress), environment, and time. Usually neglected are the effects of electric and magnetic fields. However, in many cases such fields can exert a significant influence, especially when acting in conjunction with the more common parameters [1,2]. Although many such effects have been presented in the scientific and technical literature, the mechanisms by which they occur are generally not well understood and moreover, the significance of the effects have not found the anticipated recognition by scientists and engineers, nor application in industry.

An ARO-sponsored workshop reviewed the effects of electric and magnetic fields on processing and properties of materials in 1989 [2]. Since then there have appeared a good number of additional papers in the literature on the subject. Although these suggest numerous potential applications, their employment by industry has been sparse. One reason for this is that the subject is not well known, nor understood. It therefore appeared desirable to hold a second workshop on the subject with the objectives to further evaluate: (a) the nature of the effects of electric and magnetic fields in materials science and engineering, (b) the mechanisms responsible for the effects, (c) the potential technological applications and (d) the limitations to these applications. The findings of the workshop would inform the scientific community and industry the status of the subject and would provide guidelines for ARO's future support of research in this area. To meet the above-listed objectives ARO sponsored the second workshop on this topic (having the title of this report), which was held at Saint Christopher Conference Center, Johns Island, South Carolina on May 16-19, 1999. Thirty-four (34) scientists and engineers from academia, government and industry representing seven (7) countries attended the workshop, presenting papers, participating in discussions and providing critical evaluations and recommendations. The names and addresses of the participants are given in Appendix I to this report.

2. Program and Presentations

The detailed program of the workshop is given in Appendix II to this report. Abstracts of the twenty-seven (27) papers by authors from six (6) countries are presented in Appendix III in alphabetical order of the speakers. The papers cover the range comprising synthesis, processing and properties, and include characterization of the effects, basic considerations, and potential applications. Arrangements have been made with *Materials Science and Engineering* to publish the full-length papers in a special issue. We are presently in the process of having the papers reviewed to ensure meeting the high standards of the journal.

3. Discussions and Recommendations

Active discussions took place following presentation of each paper, in special sessions and informally throughout the entire workshop. The following comprised the major comments of a general nature:

- (1) The degree and extent of the effects of electric and magnetic fields on materials science and engineering was greater than many had realized.
- (2) Very little is known regarding the basic mechanisms by which these effects occur. They should be investigated using the most advanced theoretical, analytical, and computational techniques.
- (3) There is need for more interaction and coupling between the various research workers in this general field, since there exist many common phenomena, basic principles and problems.
- (4) The materials industry has been reluctant to use electric and magnetic fields in production processes because of: (a) their unfamiliarity with such fields in materials processing and (b) there exist insufficient data on the benefits and cost effectiveness of employing such fields.

Some of the more important research activities recommended by the workshop participants, especially for overcoming the problems listed in items 2-4, above included:

A. Synthesis, Sintering and Joining.

A-1. Basic Studies

- a. What is the nature of field effects at interparticle contacts, e.g. does a plasma exist?
- b. Does activation of powder surfaces occur during electrocompaction and electric field-assisted sintering? Included are issues of surface heating, ionization, charge transfer, recombination, sputtering of atoms from the powder surface.
- c. Anisotropic effects of the field need to be considered.
- d. The roles of electromigration and electroplasticity need to be determined.

A-2. Microstructure and Experiments

- a. Detailed, advanced and high resolution microscopy studies should be carried out to determine structural integrity and other evidence of changes produced by the field.
- b. Elementary sintering process with and without field should be investigated.

A-3 Modeling

- a. Field effects should be modeled through a collection of contacting particles and include arcing, discharge, constriction, resistance, transients, electromigration, electroplasticity, and heat conduction. Experiments should be conducted to validate the models.

B. Phase Transformations, Working and Forming, and Properties

B-1 Basic Studies

- a. Determine the values of the effective valence Z^* in electromigration or field-assisted diffusion. The values are not well known except for the common FCC metals.
- b. Determine the dielectric constants of pertinent materials as a function of temperature and frequency. Only little data are available.

c. Calculate (and measure) the space charges at interfaces and dislocations in halides and oxides and their influence on diffusion. Include the interaction of charged and trapped defects with the field.

d. Calculate the effect of electric field and/or current on the activation energy for dislocation-crystal defect interactions and on the activation energy for cross slip in halides and ceramics.

e. Calculate the effect of a field on the free energy of pertinent phases.

f. Investigate the charge on vacancies in metals.

B-2 Microstructure and Experiments

a. Perform advanced, high resolution microscopy studies on the nucleation and growth of phases, and also on dislocation structure and mobility.

b. Investigate the mechanical behavior in single crystals and with well-controlled surface conditions.

c. Investigate the influence of the frequency of the field compared to the jump frequency of the atoms and/or vacancies. Also, consideration should be given to the wave form, e.g. square wave or sine wave.

B-3 Modeling

a. Develop models for the various phenomena and check their validity by experiments.

C. General Comments and Recommendations

1. Electric and magnetic fields offer considerable potential for preparing new materials and processing difficult materials. The joining of materials is one area of considerable promise.

2. Joule heating and other side effects must be separated out in the evaluation of the effects of electric and magnetic fields *per se*.

3. In view of their importance, recommended candidate materials are amorphous, nanocrystalline and metastable ceramics, intermetallic

compounds and composites. However, the more conventional metals and alloys should not be ignored. Special attention should also be given to thin films, coatings, layered structures and composites.

4. Electric and magnetic fields offer considerable potential in the design and development of new and innovative fabrication techniques for architected structures ranging from the atomic through the microstructure and mesostructure scales to the macroscale.

5. There should be more interaction, cooperation, and coupling between the various investigators working in this general field. Strongly encouraged was the formation of a Center with participation of the investigators attending the workshop and representing a number of universities, activities and disciplines. Prof. H. Conrad agreed to provide the leadership for preparing a proposal to NSF to establish such a Center.

4. References

1. H. Conrad, "Influence of Electric Currents and Fields on the Kinetics and Microstructure of Phase Transformations in Metals and Ceramics," Final Report ARO Res. Agreement DAAH04-94-G-0311, October 15, 1997.
2. H. Conrad and I. Ahmed, ARO Workshop "High Intensity Electro-Magnetic and Ultrasonic Effects on Inorganic Materials Behavior and Processing," N. C. State University, July 17-18, 1998.

Appendix I

ARO WORKSHOP PARTICIPANTS DIRECTORY

Antolovich, Stephen

Mechanical and Materials Engineering

Washington State University

Pullman, WA 99164-2920

Phone: (509)335-8654

Fax:

E-Mail:

Baranov, Yury V. (Working and Forming)

Mechanical Engineering Research Institute

Russian Academy of Sciences

101830 Moscow Centre

M. Kharitonyevsky per., 4

Moscow, Russia

Phone: (7-095)925 37 82 (7-095)924 88 70

Fax: (7-095)200 42 39 (7-095)135 77 69

E-mail:

Byeon, Soon Cheon (Sintering and Joining)

Research Institute of Advanced Materials

Seoul National University

Shillimdong, Kwanacku

Seoul 151-742

Korea

Phone: +82-2-880-8316

Fax: +82-2-886-4156

E-mail: byeon@plazal.snu.ac.kr

Present Temporary Address

205 Bevill Research Bldg.

University of Alabama

PO Box 870209

Tuscaloosa, AL 35487

Phone: (205)348-2738

Fax: (205)348-2346

E-mail: sbyeon@magnet.mint.ua.edu

Appendix I (Cont'd)

Campbell, Jim

Materials Science & Engineering Department
North Carolina State University
Raleigh, NC 27695-7907
Phone: (919)515-7278
Fax: (919)515-7724
E-mail:

Clark, David E. (Synthesis)

Department of Materials Science & Engineering
University of Florida
Gainesville, FL 32611
Phone: (352)392-7660
Fax: (352)392-3163
E-mail: dclar@mse.ufl.edu

Conrad, Hans (Working and Forming, Solid State Transformation)

Materials Science & Engineering Department
North Carolina State University
Raleigh, NC 27695-7907
Phone: (919)515-7443
Fax: (919)515-7724
E-mail: hans_conrad@ncsu.edu

Daily, John W. (Synthesis)

Mechanical Engineering Department
University of Colorado
Campus Box 427
Boulder, CO 80309-0427
Phone: (303)492-7110
Fax:
E-mail: john.daily@colorado.edu

Dolinsky, Yuli (Basic Considerations)

Department of Mechanical Engineering
Ben-Gurion University of the Negev
PO Box 653, Beer-Sheva 84105
Israel
Phone: + 972-76477078
Fax: + 972-76472813
E-mail: elperin@menix.bgu.ac.il

Appendix I (Cont'd)

Fahmy, Yusef (Melting and Casting, Properties and Durability)
Materials Science & Engineering Department
North Carolina State University
Raleigh, NC 27695-7907
Phone: (919)515-7278
Fax: (919)515-7724
E-mail: yfahmy@unity.ncsu.edu

Galligan, James (Basic Considerations)
Institute of Materials Science
University of Connecticut
Storrs, CT 06268
Phone: (203)486-3541
Fax:
E-mail:

Gillon, Pascale (Processing)
EPM-Matformag
Centre National des Recherches Scientific
25 Avenue des Martyrs BP166
38042 Grenoble Cedex 9
France
Phone: 04 76 88 90 34
Fax: 33 76 88 12 80
E-mail: gillon@labs.polycnrs-gre.fr

Gilman, John J. (Working and Forming)
6532 Bolter Hall
University of California Los Angeles
Los Angeles, CA 90024
Phone:
Fax: (310)-206-7353
E-mail: gilman@seas.ucla.edu

Groza, Joanna (Sintering and Joining)
Department of Chemical Engineering & Materials Science
University of California Davis
Davis, CA 95616-5294
Phone: (530)752-8825
Fax: (530)752-9554
E-mail:

Appendix I (Cont'd)

Gschneider, Jr., Karl A. (Properties and Durability)

Ames Laboratory

Iowa State University

Ames, Iowa 50011-3020

Phone: (515)294-7931

Fax: (515)294-9579

E-mail: cagey@ameslab.gov

Koch, Carl C. (Solid State Transformation)

Materials Science & Engineering Department

North Carolina State University

Raleigh, Nc 27695-7907

Phone: (919)515-7340

Fax: (919)515-7724

E-mail: carl_koch@ncsu.edu

Lambert, Etienne R.

Assoc. Vice President Technology

Bekaert Technology Center

N.V. Kekaert S.A., Bekaertstraat 2

B-8550 Belgium

Phone: + 32 56 76 71 57

Fax: + 32 56 76 70 20

E-mail: lambert_etienne@btc.bekaert.com

Lavery, John

Mathematical and Computer Sciences Division

U.S. Army Research Office

P.O. Box 12211

Research Triangle Park, NC 27709-2211

Phone: (919)549-4253

Fax: (919)549-4354

E-mail: lavery@aro-emh1.army.mil

Li, James C. M. (Basic Considerations)

Department of Mechanical Engineering/Materials Science Program

233 Hopeman/River Campus

Rochester, NY 14627

Phone: (716)275-4038

Fax: (716)256-2509

E-mail: li@me.rochester.edu

Appendix I (Cont'd)

MacDonald, Bruce

Program Director Metals Research
National Science Foundation
Division of Materials Research, Room 1065
4201 Wilson Blvd.
Arlington, VA 22230
Phone: (703)306-1835
Fax: (703)306-0515
E-mail: bmacdona@nsf.gov

Molotskii, Michel I. (Basic Considerations)

School of Physics and Astronomy
Tel Aviv University
Tel Aviv 69978
Israel
Phone:
Fax:
E-mail: molot@internet-zahav.net

Mukherjee, Amiya K. (Sintering and Joining)

Department of Chemical Engineering & Materials Science
University of California Davis
Davis, CA 95616-5294
Phone: (530)752-1776
Fax: (530)752-9554
E-mail: akmukherjee@ucdavis.edu

Mullins, William M.

U.S. Army Research Office
AMSRL-RO-PM
PO Box 12211
Research Triangle Park, NC 27709-2211
Phone: (919)549-4286
Fax: (919)549-4399
E-mail: mullinsw@aro-emh1.army.mil

Munir, Zuhair (Synthesis)

Department of Chemical Engineering & Materials Science
University of California Davis
Davis, CA 95616
Phone: 530-752-5132
Fax: 530-752-9554
E-mail: zamunir@ucdavis.edu

Appendix I (Cont'd)

Newman, Duane C. (Sintering and Joining)

IAP Research, Inc.

2763 Culver Avenue

Dayton, OH 45429-3723

Phone: (937)296-1806

Fax: (937)296-1114

E-mail: dnewman@iap.com

Okazaki, Kenji (Sintering and Joining)

Chemical and Materials Engineering Department

177 Anderson Hall

University of Kentucky

Lexington, KY 40506-0046

Phone: (606)257-1307

Fax: (606)323-1929

E-mail: egrulke@engr.uky.edu

Omori, Mamoru (Sintering and Joining)

Institute for Materials Research

Tohoku University

2-1-1 Katahira, Aoba-Ku

Sendai 980-8577

Japan

Phone: + 81-22-215-2106

Fax: + 81-22-215-2107

E-mail: mamori@imr.tohoku.ac.jp

Raj, Rishi (Basic Considerations)

Department of Mechanical Engineering

University of Colorado

Boulder, CO 80309-0427

Phone: (303)492-1029

Fax: (303)492-3498

E-mail: rishi.raj@colorado.edu

Rath, Bhakta, B.

Materials Science and Component Technology Directorate, Code 6000

Naval Research Laboratory

4555 Overlook Avenue, S.W.

Washington, DC 20375-5341

Phone: (202)767-3566

Fax: (202)404-1207

E-mail: rath@utopia.nrl.navy.mil

Appendix I (Cont'd)

Shang, Jian-Ku (Properties and Durability)
Department of Materials Science and Engineering
University of Illinois at Urbana-Champaign
1304 West Green Street
Urbana, IL 61801
Phone:
Fax:
E-mail:

Simmons, Wilbur
1012 Terrapin Crossing Rd.
Liberty, SC 29657
Phone: (864)868-7893
Fax:
E-mail: suowwism@mindspring.com

Spontak, Richard J. (Properties and Durability)
Materials Science & Engineering Department
North Carolina State University
Raleigh, NC 27695-7907
Phone: (919)515-4200
Fax: (919)515-7724
E-mail: spontak@mat.mte.ncsu.edu

Stadelmaier, Hans (Basic Considerations)
Department of Materials Science & Engineering
North Carolina State University
Raleigh, NC 27695-7907
Phone: (919)515-2349
Fax: (919)515-7724
E-mail: stadelmaier@ncsu.edu

Troitsky, Oleg.A. (Working and Forming)
Firm Troyka and Russian Academy of Sciences
4 General Antonov Street
Bldg. 2, Apt. 152
117279 Moscow, Russia
Phone:
Fax: (095)334-7114
E-mail:

Appendix I (Cont'd)

Yang, Di

Materials Science & Engineering Department

North Carolina State University

Raleigh, NC 27695-7907

Phone: (919)515-7278

Fax: (919)515-7724

E-mail:

Appendix II

Program for ARO Workshop

Enhanced Synthesis, Processing and Properties of Materials
with Electric and Magnetic Fields
Saint Christopher Conference Center
Johns Island, South Carolina

Sunday, May 16, 1999

5:00 - 8:00 p.m.	Registration
6:00 - 7:00 p.m.	Social
7:00 p.m.	Dinner

Monday, May 17, 1999

7:15 - 7:45 a.m.	Breakfast
8:00 - 8:05 a.m.	Welcome: H. Conrad, N. C. State University
8:05 - 8:30 a.m.	<i>Introduction and Rationale for Workshop:</i> W. Simmons, ARO

I. Basic Considerations: Chairman - J. Gilman, University of California, Los Angeles

8:30 - 9:00 a.m.	Charged Dislocations in Ionic Crystals J. C. M. Li, University of Rochester
9:00 - 9:30 a.m.	Space Charge Controlled Diffusional Creep R. Raj, University of Colorado
9:30 - 10:00 a.m.	Magnetic Properties of Materials H. Stadelmaier, North Carolina State University
10:00 - 10:15 a.m.	Coffee
10:15 - 10:45 a.m.	Measurement of Instantaneous Dislocation Velocities During Tensile Testing J. Galligan, University of Connecticut
10:45 - 11:15 a.m.	Theoretical Basis for Electro- and Magneto- Plasticity M. Molotskii, Tel Aviv University

Appendix II (Cont'd)

11:15 - 11:45 a.m. Peculiarities of Coexistence of Phases with Different
Electrical Conductivities Under the Influence of Electric
Current

Yuli Dolinsky, Ben-Gurion University

11:45 - 12:00 noon General Discussion: Is the Basic Science in Good Shape?

12:00 - 1:00 p.m. Lunch

II. Synthesis: Chairman - Z. A. Munir, University of California, Davis

1:00 - 1:30 p.m. Electric Field Control of Particle Formation and Deposition in
Gas Phase Synthesis

J. Daily, University of Colorado

1:30 - 2:00 p.m. Electric Field Activated Combustion Synthesis

Z. A. Munir, University of California, Davis

2:00 - 2:30 p.m. Microwave Activated Synthesis

D. E. Clark, University of Florida

2:30 - 2:45 p.m. *Discussion: Synthesis* - Benefits vs Difficulties and Costs

2:45 - 3:00 p.m. Coffee

III. Processing: Chairman - C. Koch, North Carolina State University

3:00 - 3:30 p.m. Use of Intense D.C. Magnetic Fields in Materials Processing
Pascale Gillon, Centre National des Rescherches Scientific,
France

III.1. Melting and Casting

3:30 - 4:00 p.m. Effects of Electric and Magnetic Fields on the Microstructure
and Segregation in Castings

Y. Fahmy and H. Conrad, North Carolina State University

III.2. Sintering and Joining

4:00 - 4:30 p.m. Novel Use of Electric Fields and Electric Currents in Powder
Metallurgy (P/M) Processing

D. Newman, IAP, Inc.

Appendix II (Cont'd)

- 4:30 - 5:00 p.m. Electric Field Assisted Bonding of Ceramics
 C. Byeon, Seoul National University
- 5:00 - 5:15 p.m. *Discussion: Processing - Benefits vs Difficulties and Costs*
- 6:00 - 8:00 p.m. Dinner
 After dinner speaker: Dr. Bhatka Rath, Naval Research
 Laboratories

Tuesday, May 18, 1999

- 7:15 - 7:45 a.m. Breakfast

III.2. Sintering and Joining (continued)

- 8:00 - 8:30 a.m. Electro-Discharge Consolidation Applied to Nanocrystalline
 and RSP Powders
 K. Okazaki, University of Kentucky
- 8:30 - 9:00 a.m. Sintering, Consolidations and Chemical Reactions by Spark
 Plasma System (SPS)
 M. Omori, Tohoku University
- 9:00 - 9:20 a.m. Electric Field Assisted Processing of Ultrafine Grained
 Alumina Matrix Composites
 A. K. Mukherjee, University of California, Davis
- 9:20 - 9:40 a.m. Sintering Activation by External Electric Field
 J. R. Groza, University of California, Davis
- 9:40 - 10:20 a.m. Microwave Sintering and Joining
 D. E. Clark, University of Florida
- 10:10 - 10:20 a.m. *Discussion - Sintering and Joining: Benefits vs Difficulties
 and Costs*
- 10:20 - 10:30 a.m. Coffee

III. 3. Working and Forming Including Superplasticity: Chairman - R. Raj, University of Colorado

- 10:30 - 11:00 a.m. Electro-plastic Effects in Semiconductors
 J. Gilman, University of California, Los Angeles

Appendix II (Cont'd)

- 11:00 - 11:30 a.m. Electroplasticity of Metals and Ceramics
 H. Conrad, North Carolina State University
- 11:30 - 12:00 noon Electric Fields and Currents in the Processing and Properties
 of Metals
 Y. Baranov, Russian Academy of Sciences
- 12:00 - 1:00 p.m. Lunch
- 1:00 - 1:30 p.m. Electroplasticity of Metals
 O. A. Troitskii, Russian Academy of Sciences
- 1:30 - 1:45 p.m. *Discussion: Working and Forming: Benefits vs Difficulties
 and Costs*
- III. 4. Solid State Transformations: Chairman - Yuli Dolinsky, Ben-
 Gurion University**
- 1:45 - 2:15 p.m. Experimental Evidence for Magnetic or Electric Field Effects
 on Phase Transformations
 C. Koch, North Carolina State University
- 2:15 - 2:45 p.m. Electric Current and Solid State Phase Transformations
 H. Conrad, North Carolina State University
- 2:45 - 3:00 p.m. *Discussion: Solid State Transformations: Benefits vs
 Difficulties and Costs*
- 3:00 - 3:15 p.m. Coffee
- IV. Properties and Durability (Life): Chairman - J. Galligan,
 University of Connecticut**
- 3:15 - 3:45 p.m. The Influence of Magnetic Fields on the Thermal Properties
 of Solids
 K. Gschneider, Iowa State University
- 3:45 - 4:15 p.m. Effects of Magnetopulsing on Mechanical Properties of Steels
 Y. Fahmy, North Carolina State University
- 4:15 - 4:45 p.m. Influence of Electric Field on Crack Growth in Ceramics
 Jian-ku Shang, University of Illinois

Appendix II (Cont'd)

4:45 - 5:15 p.m. Electric- and Magnetic-Field Responsive Polymers: An Overview
 R. Spontak, North Carolina State University

6:00 - 8:00 p.m. Dinner

Wednesday, May 19, 1999

7:15 - 7:45 a.m. Breakfast

8:00 - 8:15 a.m. *Discussion: Properties and Durability: Benefits vs Difficulties and Costs*

8:15 - 8:30 a.m. *General Comments:* H. Conrad, North Carolina State University

V. Discussions: Status, Applications and Future Research

8:30 - 10:00 a.m. Individual Topic Group Discussions:
 Chairmen - Respective Chairs of Topics, Yuli Dolensky,
 T. Elperin, J. Galligan, J. Gilman, C. Koch, Z. Munir, R. Raj

(a) How significant are the effects? Magnitude and costs.

(b) Potential commercial applications.

(c) Future research: basic and applied.

10:00 - 10:30 a.m. Coffee

10:30 - 11:30 a.m. *General Discussion: Present Status and Future*
 H. Conrad and W. Simmons, Co-Chair
 Panel: T. Elperin, J. Galligan, J. Gilman, C. Koch, Z. Munir,
 R. Raj

11:30 - 12:30 p.m. LUNCH

1:00 p.m. End of Workshop

Alloys Deformation Features in Electric Static Fields

Yu. V. Baranov

Mechanical Engineering Research Institute
(Moscow, Russia)

Work on both distribution of electron density near metal surface is considered and impact on it of exterior electric field. Results on definition of surface barrier on the border of metal-vacuum, metal-dielectric in exterior electric static field are analyzed. Penetration of electric field into metal and its influence on value of surface barrier is considered.

Influence of electric static field with stress from 0 up to 7.0×10^6 B/M on mechanical properties of nickel (N3), brass (L62), steel (S30), stainless steel (12X18HI0T) at tension with deformation speeds $=1.25 \times 10^3$ and 2.0×10^4 c^{-1} has been analyzed on the installation for comprehensive physical and mechanical research (IMASH-20-78/ ALA-TOO) in vacuum 10^{-5} Torr. It has been ascertained that:

- 1) electric static field causes hardening of metallic samples, which is characterized by more high stress of a flow as compared to deformation without field
- 2) value of hardening grows with the strain increase of electric static field (E), and maximum deformation at fracture decreases
- 3) creation of a negative potential at the sample's surface leads to larger hardening
- 4) effect decreases with increase of deformation speed
- 5) effect is greater in plastic metals
- 6) deformation in the field doesn't require more power consumption than regular deformation
- 7) deformation of surface layers with thickness less than 10mcm in a negative field causes appearance of a «tooth» of yield, which is connected, perhaps, with electron and dislocation interactions in a surface layer
- 8) yield limit of surface layers (S_y) depends on a layer's thickness and on direction of electric static field. Yield limit of surface layers decreases with growth of layer's thickness, aspiring to an ordinary value of a sample's yield limit (S_y).
- 9) deformation of nickel (N3), brass (L62), steel (S30), stainless steel (12X18HI0T) in electric static field causes friction force increase of crystalline lattice, and with deformation speed increase, friction force decreases
- 10) deformation in electric static field facilitates destruction suppression; with application of the field (E), destruction coefficient (Δ) decreases and durability coefficient (ζ) increases; deformation speed decrease is increasing this effect
- 11) for metals with a greater Yung module change of Δ and of ζ in the field is lesser, than for plastic metals
- 12) critic stress (σ_c) which causes appearance of first micro-cracks in metals deformed in electric static field, is larger than during deformation without field; critic stress (σ_c) is larger in negative fields.

Appendix III (Cont'd)

New Electroimpulse Current Technologies of Treatment of Tools and Steels for Obtaining Unique Properties of Materials

Y.V. Baranov
Mechanical Engineering Research Institute
(Moscow, Russia)

Basing on experimental and theoretic research of deformation and fracture processes of metals and alloys, physical laws of materials behaviour under deformation have been determined:

- a) in polishing media;
- b) under electric current impact;
- c) under pulse electric current impact ($j_{\max} = 800 \text{ A/mm}^2, \Delta \tau = 0,02 \text{ sec}, V = 6-12 \text{ Volt}$).

New treatment technologies for metals and alloys have been offered using determined effects under drawing, rolling and bending deformation.

- a) Taking an example of tungsten monocrystals of electron beam zone melting, it is shown that their deformation in a polishing medium (NaOH-2% solution in water, $V = 10 \text{ Volt}$, $j = 1 \text{ a/sm}^2$) makes it possible to increase maximum deformation meaning up to fracture 8 times as much; and in this case deformation tension is decreased substantially.

Basing at this effect of metals plastification in polishing media, technologies of deformation of a wide circle of metals and alloys by bending have been developed (for instance - Al-, Ti-based alloys, of different steels).

- b) It has been experimentally proven, that coming of pulse electric current through deformed metal substantially increases its plasticity. The given effect was named electric plastic effect (1968, O.A. Troitsky). The given effect is widely used while wire drawing. At present a rolling mill is being developed using electric plastic effect.
 - c) It has been experimentally proven that metal-cutting instrument treatment (of drills, etc.) by pulse electric current increases durability of the instrument during cutting 3.5 times as much. Basing on this effect a technology of durability increase of drills of different diameter (up to 20 mm) using pulse current treatment has been developed.
- 2) It has been experimentally and theoretically proven that pulse current treatment makes it possible to heel structural defects in metals and alloys (porosity, microporosity, microcracks, etc.) which appear under course of operation of the items made of this material. Basing on this effect modes of defect healing by pulse current have been offered for stainless, structural and instrumental steel.

Appendix III (Cont'd)

Electric Field Assisted Bonding of Ceramics

Soon Cheon Byeon and Kug Sun Hong*

Center for Materials for Information Technology, 205 Bevill Research Bldg., P.O. Box 870209, Univ. of Alabama, Tuscaloosa, AL 35487-0209

*Research Institute of Advanced Materials, College of Engineering, Seoul National University, Seoul, 151-742, KOREA

Bonding between oxide ceramics using an electric field was reviewed. There is a general prerequisite for an effective bonding using an electric field. Only cations should be moved effectively by an applied electric field. If anions can be transported effectively by an applied electric field there is no bonding. The lattice near anode annihilated and the lattice was newly made near cathode. The microstructure near anode is significantly different from that near cathode.

For a given temperature, bonding between manganese-zinc ferrites was accelerated with the application of an electric field. The degrees of bonding increased remarkably as the bonding time and bonding temperature increased. Moreover, the direction of an applied current should be from the poly- to single-crystalline ceramics regions in order to enhance bonding effectively. These phenomena can be applied to the preparation of large single crystal cost effectively.

Above 1100°C the effect of electric field was not obvious due to thermally activated self-motion of atoms. Bonding between manganese-zinc ferrites improved with increased oxygen partial pressure. This was attributed to the higher diffusion coefficient of the cations at higher oxygen partial pressures. The orientation of the single crystal affected not only the bonding process itself but also influenced morphological changes in the pores at the bonded interface. The bonding was more efficient when the configuration of the bonding planes was symmetrical rather than when the configuration was nonsymmetrical. The shape of pores at the bonded interface was also dependent on the orientation of the crystal. The interface and the pores were not changed when the orientation of the crystal remained constant.

Appendix III (Cont'd)

Microwave-Activated Synthesis

David E. Clark
University of Florida
Dept. of Materials Science & Engineering
Gainesville, FL 32611-6400

Although the major uses of microwave energy are communication and radar, its use for processing materials is emerging rapidly. Presently, microwave energy is used on a large scale in food preparation, vulcanization of rubber and manufacturing of polymer/wood composites. These processes take advantage of the selective heating, large depth of penetration of rapid heating that microwave energy provides in comparison to conventional heating methods. During the last 15 years, researchers at universities, national laboratories and industry have demonstrated that microwave energy can be used to process many types of materials, including organics, ceramics, polymers, glasses, sol-gels, metals and composites. In some cases, the processing rates are significantly greater than with conventional methods, while in other cases, the properties of the microwave-processed materials are superior.

At the University of Florida, a number of materials processes have been under investigation since 1986. For this talk, several examples which illustrate the potential applications for microwave processing have been selected, including: microwave joining and repair; microwave-initiated and controlled combustion synthesis; microwave sintering of armor materials; microwave nucleation and crystallization in glass.

Appendix III (Cont'd)

ELECTROPLASTICITY IN METALS AND CERAMICS

Hans Conrad
Materials Science and Engineering Department
North Carolina State University
Raleigh, N. C. 27695-7907

Abstract

In the case of *metals*, the following are considered: (a) the effects of a high density electric current pulse and (b) the effects of an external electric field on superplasticity. The major effect of the current pulse was to reduce the thermal component of the flow stress, which resulted from the combined action of: (a) an electron wind force, (b) a decrease in the activation enthalpy and (c) an increase in the pre-exponential, the last making the largest contribution. Besides giving a reduction in the flow stress (10-20%), an external electric field reduced cavitation and grain growth during superplastic deformation. The influence of the external field appears to be on the migration of charged vacancies along grain boundaries.

In the case of *ceramics*, the effects of an internal electric field on the following are considered: (a) the plastic deformation of polycrystalline NaCl at $0.28-0.75 T_M$ and (b) the superplasticity of fine-grained oxides (MgO , Al_2O_3 and ZrO_2) at $T > 0.5 T_M$. Regarding NaCl, at $T \leq 0.5 T_M$ an electric field $E \geq 10 \text{ kV/cm}$ was needed to enhance dislocation mobility in single crystals. In contrast, a field of only $E \leq 1 \text{ kV/cm}$ enhanced cross slip in polycrystals, thereby significantly reducing the flow stress. At $T > 0.5 T_M$, there occurred a decrease in the flow stress of polycrystalline NaCl through a reduction in the rate-controlling diffusion activation energy. Regarding the fine-grained oxides at $T > 0.5 T_M$, an internal electric field $E \leq 0.3 \text{ kV/cm}$ gave an appreciable (50-80%), reversible reduction in the flow stress by an enhancement of the rate-controlling diffusion process. Limited work suggests that the field may also retard grain growth and cavitation.

Appendix III (Cont'd)

EFFECTS OF ELECTRIC CURRENT ON SOLID STATE PHASE TRANSFORMATIONS

Hans Conrad

Materials Science and Engineering Department
North Carolina State University
Raleigh, N.C. 27695-7907

Abstract

The influence of an electric current on the following solid state transformations in metals are considered: (a) intermetallic compound formation and growth in diffusion couples, (b) precipitation, (c) crystallization of amorphous alloys and (d) recrystallization and grain growth of cold worked metals. The formation and growth of intermetallic compounds was to some degree in accord with electromigration theory. Regarding precipitation, in Al alloys an electric current could either enhance or retard the precipitation rate, depending on the alloy composition and the current frequency. An important factor appears to be the effect of current on the quenched-in vacancies. Both a continuous dc current and high current density electropulsing enhanced the crystallization rate of amorphous alloys. The effects were greater than could be explained by simple electromigration theory, and indicate the cooperative motion of a large number of atoms. Electropulsing enhanced the recrystallization rate of cold worked metals, but retarded subsequent grain growth. Enhancement of the recrystallization rate resulted mainly from an increase in the pre-exponential factor of the Arrhenius rate equation. Retardation of subsequent grain growth resulted from a lower residual dislocation density within the newly formed grains.

Appendix III (Cont'd)

John W. Daily

Department of Mechanical Engineering ECME 224

Center for Combustion and Environmental Research

University of Colorado at Boulder

Electric Field Control of Particle Formation and Deposition in Gas Phase Synthesis

Because of the small mass of particles synthesized in the gas phase, electrophoretic effects can be quite large and offer a means for control of growth and deposition rates. The talks will discuss the physics of particle charging, possibilities for control of growth rates, and describe research in the use of electric fields to enhance deposition rates in the manufacture of silica boules for optical fiber manufacturing.

Appendix III (Cont'd)

Peculiarities of Coexistence of Phases with Different Electric Conductivities Under the Influence of Electric Current

Yu. Dolinsky and T. Elperin

The Pearlstone Center for Aeronautical Engineering Studies

Department of Mechanical Engineering

Ben-Gurion University of the Negev

P. O. Box 653, Beer-Sheva 84105

ISRAEL

During first-order phase transitions in current-carrying conductors configuration of the electric current changes. The latter results in the appearance of the ponderomotive forces which may prevent or promote this change of the configuration of electric current and, therefore, prevent or promote a phase transition.

We investigated the influence of the ponderomotive forces caused by a change of electric current configuration on the dynamics of phase transitions and established the existence of the following effects: 1) Splitting of the curve of phase equilibrium into two different curves for the direct and inverse phase transitions; 2) Occurrence of the domain of simultaneous metastability of both phases where the direct and the inverse phase transitions proceed simultaneously; 3) Effect of ponderomotive forces on the dynamics of phase transitions is different for the volumetric and the surface phase transitions. In the case of a volumetric phase transition ponderomotive forces prevent formation of the high-temperature phase with a low conductivity and promote formation of a low-temperature phase with a higher electric conductivity. In the case of a surface phase transition at the initial stage the ponderomotive forces prevent formation of the low-temperature phase with a higher electric conductivity and promote formation of a high-temperature phase with a lower electric conductivity.

Most of these results were obtained using two different but complementary models, namely, a discrete Doering-Volmer-Zeldovich model and a continuous Ginsburg-Landau model

Appendix III (Cont'd)

EFFECTS OF ELECTRIC AND MAGNETIC FIELDS ON THE MICROSTRUCTURE AND SEGREGATION IN CASTINGS

Yusef Fahmy and Hans Conrad
Materials Science and Engineering Department
North Carolina State University
Raleigh, NC 27695-7907

ABSTRACT

Examples of the influence of electric and magnetic fields on the microstructure and segregation in castings of metals, semiconductors and polymers is presented. Both a low density continuous DC electric current and high density electropulses applied during the solidification process have been reported to refine the microstructure of metal alloy castings. Also, high density electropulses applied to the liquid prior to casting altered the microstructure of the steel castings. A theoretical model developed by Qin and Zhou is in qualitative accord with the refinement in grain size produced by electropulsing during solidification.

The rate of epitaxial growth of GaAs crystals from the melt was enhanced by the passage of a DC current and their perfection improved. A theoretical model developed for the behavior is in good accord with experiment. Single crystals of camphor could be grown from a solution of camphor in CCl_4 by applying an electric field across the surface of the melt.

Application of a magnetic field during the directional solidification of hypoeutectic Pb-Sn alloys caused severe distortion in the cellular array morphology with extensive channel formation in the mushy zone due to thermosolutal convection. A magnetic field applied during directional solidification of metal alloys gives a Lorentz force which opposes the natural solutal buoyancy force. The simultaneous application of electric and magnetic fields during solidification of Al-Si alloys produces electromagnetic vibrations, which gave an increase in number and decrease in size of the suspended Si particles at temperatures higher than the liquidus. An electromagnetic induced cavitation phenomena was responsible for crushing the particles.

Appendix III (Cont'd)

EFFECTS OF MAGNETOPULSING ON MECHANICAL PROPERTIES OF STEELS

Yusef Fahmy
Materials Science and Engineering Department
North Carolina State University
Raleigh, NC 27695-7907

ABSTRACT

Tool steels have been shown to exhibit reduced fracture toughness and fatigue life when tested in a continuous or sustained magnetic field. Pulsed magnetic fields, however, have been observed to improve fatigue and tool life of many steel alloys. The degree of improvement is found to depend on the amount of damage (fatigue or cold work) prior to application of the pulsed magnetic field treatment. The mechanism responsible for such behavior is believed to involve dislocation interaction with domain walls and relief of internal stresses.

Appendix III (Cont'd)

Measurement of Instantaneous Dislocation Velocities During Tensile Testing

J.M. Galligan

Department of Metallurgy and Metallurgical Engineering
University of Connecticut
Storrs, CT 06269-3136

Dislocation motion is affected by the electrons of a metal; the electron's trajectory can be altered by a magnetic field, leading to a change in stress for plastic deformation. By sweeping a magnetic field through the plane in which dislocations are moving the electron-dislocation-interaction can be substantially enhanced. This enhancement allows the measurement of dislocation velocities, while a crystal is plastically deforming. Further, these dislocation velocities can be measured as a function parameters, including temperature, strain rate, and plastic strain.

This method has been applied to studies of the deformation of zinc crystals and iron crystals in the temperature range of ~ 1.5 to 5K, a range of temperature where if kinks are determining dislocation velocity should decrease by many orders of magnitude on going from 4.2K to 2K. The experimental results in zinc and iron are not consistent with a kink mechanism controlling dislocation velocity.

As the temperature decreases the dislocation velocity remains constant in zinc or the dislocation velocity increases with decreasing temperature in iron. These observations rule out the kink mechanism as a controlling factor in the movement of the dislocations in these materials.

Comparison with existing theories of the influence of a magnetic field on dislocation velocities will be given^(1,2,3).

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Appendix III (Cont'd)

Uses of intense d. c. magnetic fields in materials processing Pascale GILLON EPM laboratory-CNRS-GRENOBLE

Apart from classical MHD effects on liquid metals such as braking of motions, stabilization of convection or shaping of free surfaces, the intense d. c. magnetic fields available from superconducting coils open the way to new phenomena which could lead to new methods in materials processing.

Homogeneous magnetic field applied during solidification produce a texturation of the solid by magnetic orientation of crystallites forming in the liquid. Examples of magnetic processing of high Tc superconductors, permanent magnets and biological material will be given.

However, the most interesting phenomena produced by strong magnetic fields are probably those obtained in the inhomogeneous parts of the field. The resulting magnetic force related to the field gradient intensity and the material susceptibility is used in numerous configurations. We will focus the presentation on three specific applications developed recently in our research laboratory : levitation, thermomagnetic convection and phase separation.

Appendix III (Cont'd)

Prof. John Gilman
6532 Bolter Hall
University of California Los Angeles
Los Angeles, CA 90024

"Electro-plastic Effects in Semiconductors"

This paper will be a reprisal of the work done done some time ago by J. H. Westbrook and the present author on the effects of passing a small current through semiconductors on measurements of their indentation hardnesses. It will include discussion of more recent work that is reported in the literature, as well as attempts to interpret the puzzling observations.

Appendix III (Cont'd)

SINTERING ACTIVATION BY EXTERNAL ELECTRICAL FIELD

Joanna R. Groza

Dept. Chemical Engineering & Materials Science
University of California, Davis, U.S.A.

Field Assisted Sintering Technique (FAST) is a non-conventional powder consolidation method in which densification is enhanced by an electrical discharge application combined with resistance heating and pressure. Interest in FAST sintering is motivated by its ability to consolidate a large variety of powder materials to high densities in short times. Full densification of metal and ceramic powders has been achieved within minutes, with a reduced number of processing steps, no need for sintering aids and more flexibility in powder handling. Although the electrical discharge effects have not been completely elucidated, distinct surface effects created by micro-discharges have been noticed in FAST consolidated specimens such as atomically clean grain boundaries, new resistivity peaks in superconductors or ceramic sintering after multiple discharges. On-going experimental and theoretical studies to provide more quantitative insight into the relevant FAST mechanisms will be presented.

Appendix III (Cont'd)

THE INFLUENCE OF MAGNETIC FIELD ON THE THERMAL PROPERTIES OF SOLIDS

K. A. Gschneidner, Jr. and V. K. Pecharsky

Ames Laboratory and Department of
Materials Science and Engineering
Iowa State University
Ames, IA 50011-3020

The magnetocaloric effect is the response of a solid to an applied magnetic field, which is apparent as a change in its temperature. For a simple ferromagnetic material near its magnetic ordering (Curie) temperature (T_c), when a magnetic field is applied the unpaired spins ($4f$ for the lanthanides or $3d$ for iron and its neighboring metals) are aligned parallel to the magnetic field and this causes the sample to warm up. When the magnetic field is turned-off the spins tend to become random and the material cools off. The adiabatic temperature change is usually positive for a magnetic field increase and negative for a magnetic field decrease, but opposite changes are known.

Scientists have made use of the magnetocaloric effect to reach extremely low temperatures (from milli-Kelvins to micro-Kelvins) in a one step process, which is known as adiabatic demagnetization. One can use a continuous process to cool by rejecting heat during the magnetizing step of the cycle and cooling in the demagnetizing step, equivalent to the compression and decompression steps in a common gas compressor refrigerator. This is simply known as magnetic refrigeration, and it can be used to cool small (watts) or large (megawatts) loads over a wide temperature range down to as low as 4K (liquefaction of He gas) to as high as $\sim 280\text{K}$ (air conditioning) depending on the application.

During the past two years we have (1) shown that magnetic refrigeration is a viable cooling technology and (2) discovered the giant magnetocaloric materials which promises to make magnetic refrigeration even more competitive with gas cycle refrigeration.

Appendix III (Cont'd)

Experimental Evidence for Magnetic or Electric Field Effects on Phase Transformations

Carl C. Koch
Materials Science and Engineering Department
North Carolina State University
Raleigh, NC 27695

ABSTRACT

This paper will present examples of experimental evidence for the influence of magnetic or electric fields on phase transformations. Phase stability and the kinetics of phase transformations have been mainly studied with the variables of temperature and, to a lesser extent, pressure. Magnetic and electric fields can also affect the free energy, and therefore phase stability. Electric fields can also influence the kinetics of thermally activated phase transformations by their effect on atomic mobility. Examples of magnetic field effects to be discussed will include effects on phase stability which can increase the M_s temperature in Fe-based alloys, bias the antiferromagnetic to ferromagnetic transformation in Fe particles in Cu, and stabilize disordered phases in ordered Fe_3Al alloys. Magnetic fields can also affect the morphology of microstructures during phase growth by heat treatment in field. The well-known ALNICO permanent magnet materials are an important example of this. Electric fields, with or without current generation, can influence a wide variety of phase transformations. Some of the examples to be discussed include changes in cast structure, hardenability, quench aging, and age hardening in metallic alloys; cholesteric to nematic phase transformations in liquid crystalline polymers; phase transformations in ceramic and polymer ferroelectric materials; and phase separation in oxide glasses. These examples will be presented and discussed. Several promising areas for future research and potential applications will be suggested.

Appendix III (Cont'd)

Charged Dislocations in Ionic Crystals James C. M. Li, University of Rochester

Since the dislocations in ionic crystals can carry charges, dislocations can be moved by an electric field and plasticity can produce electric potential differences between specimen surfaces. The former can be used to measure charges on the dislocations and the latter can be used to estimate mobile dislocation density. These will be reported together with the mobile dislocation density in latent hardening, stress cycling, Bauschinger effect and stress relaxation.

Appendix III (Cont'd)

Theoretical Basis for Electro- and Magnetoplasticity

Michel I. Molotskii

School of Physics and Astronomy, Beverly and Raymond Sackler Faculty of Exact Science, Tel Aviv University, Tel Aviv 69978, Israel

The influence of a magnetic field on dislocations depinning from paramagnetic obstacles is studied. It is suggested that the depinning may take place due to the following mechanism. When a dislocation (or kink of dislocation) passes an obstacle the unsaturated electron bonds of the dislocation core and the obstacle form a radical pair. Magnetic field induced intercombination transitions from the binding to an antibinding state of the radical pair lead to an additional population of high spin antibinding states with lower binding energy. This results in a plasticity growth.

This model is used to develop the quantitative theory of the influence of a magnetic field on the range of a dislocations. The theory also is used to consider the influence of a magnetic field on the amplitude independent internal friction of dislocations in metals. The theory is compared with the experimental data obtained for undeformed and deformed copper. Excellent agreement is achieved in deformed metals.

The resonance enhancement of the magnetoplastic effect by additional, even weak, microwave magnetic field was predicted in our work in 1996 and actually observed in 1998. Recently we suggested that the nuclear spin may affect plasticity in magnetic fields through its hyperfine interaction with the electron spin. It is shown that the hyperfine interaction lead to a threshold type behavior of the magnetic field dependence of various plasticity related quantities. Such thresholds were really discovered in the experiments.

An explanation of the electroplastic effect is proposed. The increase of the metal plasticity is brought about by the facilitation of dislocations depinning caused by the current induced magnetic field. This mechanism allows one to explain the principal features of the effect which include its nonpolarity, characteristic value of the current density, the dependencies of the stress drop on the current, etc. Possible experiments for testing the model are discussed.

Appendix III (Cont'd)

Electric Field Assisted Processing of Ultrafine Grained Alumina Matrix Composites

by

R. S. Mishra and A. K. Mukherjee

ABSTRACT

The densification of alumina is influenced by the electrical field and the starting crystal structure of alumina powder. The application of electric pulsing enhanced the densification and the level of enhancement depends on the pulsing sequence. A multiple pulsing sequence, i.e., intermittent pulsing up to sintering temperature, leads to considerable enhancement as compared to the results obtained without any pulsing and single pulsing sequence at room temperature. The alpha alumina powders sintered more readily than the gamma alumina powders. The development of vermicular structure during the transformation of gamma alumina powders lowers the sintering kinetics. Pure alumina specimens with >98 % theoretical density were obtained in less than 10 minutes at 1300 °C. The average grain size was ~0.7µm. Similar sintering parameters were used to synthesize alumina based nanocomposites with >99 % theoretical density in 5-10 minute sintering time. The nanocomposites show enhanced fracture toughness. The electric field and pressure assisted sintering is a very effective way to synthesize nanocomposites in a short sintering time at lower temperatures.

Appendix III (Cont'd)

Electro- Discharge Consolidation Applied to Nanocrystalline and RSP Powders

Kenji Okazaki
Department of Chemical and Materials Engineering
University of Kentucky
Lexington, KY 40506-0046, U. S. A.

Electro-discharge consolidation (EDC) employs a high-voltage, high-density current pulse (up to 30 kV, 15,000 A/mm²) to powders under pressure for consolidation. Because of a short discharge time ($\sim 300 \mu\text{s}$), there are four characteristics in EDC; the removal of oxide films on the prior powder particle surface, densification, preservation/or modification of microstructures inherent in the starting powders and improvement of mechanical properties. Accordingly, this EDC technique has advantages over the conventional consolidation for nanocrystalline and RSP powders.

First, nanocrystalline powders of a 77 at.%Nb-23at.%Al were prepared by mechanical alloying. Careful deconvolution of XRD revealed that the MA powder was a mixture of multi-phase nanocrystalline (5 -9 nm) Nb₃Al, Nb₂Al, Nb and Al. The multi-phase powder under external pressure was subjected to a high-voltage, high-density current pulse to produce a consolidated bulk having a relative density of up to 99%; still a mixture of two nanocrystalline Nb₃Al (92 vol.%, 36 nm) and Nb₂Al (8 vol.%, 26 nm). These nanocrystalline bulk (<35 nm) exhibits a negative Hall-Petch relationship; grain size softening takes place. EDC bulks showed a compressive yield stress of nearly 900 MPa and 1.4% plastic ductility at ambient temperature, which were 400 MPa and 49% at 1183K with a strain rate sensitivity of 0.47, suggesting that nanocrystalline materials deform superplastically by a grain rotation mechanism.

Secondly, EDC was applied to Al₉₁Ni₅Cu₂Ti_{1.4}Zr_{0.4}Mn_{0.2} powders made by gas atomization and mechanical alloying. A prominent difference between these two is the amount of solute atoms in solid solution. EDC did not alter the microstructures inherent in atomized and MA powders. Because of high degree of precipitation in atomized powders, their bulks have higher yield and ultimate strengths and lower ductility compared to the counterpart. The magnitude of ductilities exhibited by EDC bulks clearly indicates that the oxide film on powder particles' surface has been completely removed.

Appendix III (Cont'd)

Enhanced Synthesis, Processing and Properties of Materials with Electric and Magnetic Fields

Monday, May 17th

Session III.2 Sintering and Joining

Novel Use of Electric Fields and Electric Currents In Powder Metal (P/M) Processing

**Duane C. Newman, Sr. Research Engineer
IAP Research Inc.
2763 Culver Ave.
Dayton, OH 45429**

IAP Research, Inc. has done significant work in applying electric fields and electric current to enhance traditional metal powder forming processes. Our interest in this area first began in 1995 when IAP Research collaborated with Dr. Conrad under the Advanced Research Project Agency (ARPA) Small Business Innovative Research Contract entitled "Exploitation of the Electroplastic Effect in Commercial Metal Forming Operations". Under this contract we evaluated approaches for utilizing electroplasticity effects in rolling and drawing operations and powder metal compaction. The two most promising results came from the use of electric current to enhance the green compaction density of commercial powder metals and the use of electric fields to enhance the surface porosity of powder metal compacts. In a second Phase I SBIR effort in 1996 for NSF, we again collaborated with Dr. Conrad in an effort entitled "Electric Field Sintering for Improved Surface Finish of PM Parts".

The focus of this talk will be on our experience with the use of electric fields and currents to enhance P/M compaction and sintering operations. IAP Research is actively engaged in product/process development in the P/M industry and is an innovator in applying advanced technology to develop new and improved processes for the P/M and the metal forming industry.

The talk will summarize our work with both capacitive discharge and DC Pulse discharge electro-compaction technology and electric field sintering. We have performed electro-consolidation studies on materials including powdered iron, stainless steel, aluminum and titanium. The results of compaction and sintering studies will be presented.

Appendix III (Cont'd)

Title: Sintering, Consolidations and Chemical Reactions by Spark Plasma System (SPS)

Presentation: Mamoru Omori (Dr.)

Affiliation: Institute for Materials Research, Tohoku University
2-1-1 Katahira, Aoba-ku, Sendai 980-8577, Japan

Abstract:

Spark plasma system (SPS) has been called spark plasma sintering (SPS) and plasma activated sintering (PAS). SPS is structured using the same electric source as that of a discharge machine. The direct pulse current of the discharge machine causes spark plasma of high energy. In the case of SPS the high energy plasma is not generated, because there is no gap on electric paths. Only weak plasma could be allowed to appear among powders in a graphite die. However, the plasma has not been identified directly. There are synthetic materials resulting from the effect of the plasma. The following examples are described. Aluminum metal can be sintered. Pure tungsten carbide and aluminum nitride powders become dense body without additives. Discharged patterns are obtained on the surface of the insulator of an oxide ceramics. Organic fibers are etched. Charcoal is solidified. Thermosetting polyimide is consolidated. Structure of insoluble polymonomethylsilane is rearrange into soluble one. It is not easy to solidify aluminum metal, aluminum nitride and tungsten carbide by normal sintering and hot pressing. The consolidated thermosetting polyimide and rearranged soluble polymonomethylsilane have not been obtained by other means. Charcoal reacts between organic functional groups. These evidences show that weak spark plasma is formed and promote sintering and chemical reactions. So applications of SPS are not only made on sintering metal and ceramics powders, but also on chemical reaction, joining and consolidation of polymers. We will able to find other applications of SPS with the effect of spark plasma.

Appendix III (Cont'd)

Rishi Raj*
Department of Mechanical Engineering
University of Colorado
Boulder CO 80309-0427

SPACE CHARGE CONTROLLED DIFFUSIONAL CREEP

Abstract

Electrically unbalanced segregation of charged defects to grain boundaries in ionic materials creates a space charge in regions immediately adjacent to the grain boundaries. The space charge leads to a change in the "carrier" concentration, causing significant changes in the effective self diffusivity. Phenomena such as diffusional creep which are controlled by self diffusion across interfaces can be significantly enhanced, or retarded by space charge. The retardation effect can be severe and is of technological interest in the design of creep resistant ceramic materials. The effect of space charge can also lead to non-linear behavior since the magnitude of the interfacial charge can be affected by the applied stress, causing the effective diffusion coefficient to become stress dependent. New experimental results that directly measure the interfacial charge as a function of the applied stress will be presented.

Appendix III (Cont'd)

Effects of Electric Fields on Crack Growth in Selected Dielectric Glasses and Ceramics

Jian-Ku Shang, Xiaoli Tan, Zhiqiang Xing, and Dwight Viehland

Department of Materials Science and Engineering
University of Illinois at Urbana-Champaign, Urbana, IL 61801

Abstract

Effects of electrical fields on cracking behavior of ceramics were examined in selected oxides, namely, glasses, magnesium oxide (MgO), lead zirconate titanate (PZT) and lead lanthanum zirconate titanate (PLZT). In the glass and MgO, DC electric field was found to inhibit development of the surface microcracks perpendicular to the field direction, resulting in apparent toughening of the solid. The magnitude of the electrical toughening was more pronounced in MgO than in soda-lime-silicate glass, and was dependent on the field strength. In PZT and PLZT, deflection of microcracks occurred when the electrical field was non-orthogonal to the crack plane. The deflection angle was as high as 90 degrees from the original crack-plane, depending on the relative orientation of the electrical field to the crack plane. Crack growth in PLZT under AC electric fields showed strong dependence on the mean electrical field.

Electric- and Magnetic-Field Responsive Polymers: An Overview

Richard J. Spontak

Departments of Chemical Engineering
and Materials Science & Engineering
North Carolina State University
Raleigh, North Carolina 27695

ABSTRACT

Over the past decade, tremendous interest has arisen in mesomorphic polymers that respond in controllable fashion to an externally imposed field. While numerous efforts have concentrated on the application of shear fields in an attempt to mechanically align the mesophases that form in, for example, liquid crystalline polymers and block copolymers, additional studies have successfully demonstrated that such alignment, which induces anisotropic optical and/or mechanical properties, can be achieved in some polymeric, not necessarily mesomorphic, systems through exposure to electric or magnetic fields. Such field sensitivity constitutes an obvious consideration in the fabrication of next-generation optical devices, but electric- or magnetic-field responsiveness can likewise be of critical importance in the rational design of emerging "smart" materials. In this case, the materials may stiffen, as evidenced by an abrupt increase in the elastic component of their modulus, due to sudden molecular alignment relative to the imposed field. Moreover, the extent to which the modulus increases is typically correlated with the strength of the field, indicating that the mechanical properties can be tuned by varying field strength. While an important commercial application of polymeric materials with tunable mechanical properties includes vehicular shock absorption, another example of where such property control is requisite is in the design of artificial muscle. Moreover, field-induced molecular orientation during polymer fiber spinning to produce high-tensile fibers constitutes an interesting technological alternative to the multistage spinning processes that are currently employed. Control over molecular orientation due to an imposed electric or magnetic field is not, however, limited to mechanical property enhancement. Many low-molar-mass organic systems are currently used in optical devices (e.g., liquid crystal displays, LCDs) due to their responsiveness to an electric field. Attempts to extend this technology to polymeric materials has met with some serious challenges due to the long chain-like nature of macromolecules. Recent efforts have, however, demonstrated mesomorphic polymers can be used to construct electric-responsive optical devices, such as the microphase-stabilized ferroelectric liquid crystal (MSFLC) ensemble. The objective of this work will be to provide an abbreviated survey of polymeric materials whose mechanical or optical properties can be tuned through an imposed electric or magnetic field, as well as a glimpse of where this exciting field may head as it continues to grow and evolve.

Appendix III (Cont'd)

Heads of the report

1. Role of a surface "Fermi" in electronic - plastic effect(EPE).
2. Technique of counter impulses(TCI) for study EPE.

O.A.Troitsky.

1. In the first part of the report the possible role of a surface is considered "Fermi" in electronic - plastic effect EPE in metals. The parameters are adduced Surfaces "Fermi" in metals, indicating large size EPE. Listing The theory EPE. Qualitatively is discussed a role of various "Fermi" carriers of a current - electrons and "Hole" in transfer of impulses of force and energy on a dislocation.

The role closed of a surface "Fermi" in transfers of impulses on a dislocation is appreciated. The role of small volumes and fragments of a surface "Fermi" in, indicated, is considered Processes and favorable directions of displacement of a surface "Fermi". Analysing Versions of transformation of a surface "Fermi" in a course EPE and correlation of a condition with size EPE in Edge_center cubic and hexagonal densely Packed metals, and also gear of transfer of impulses from carriers of a current To dislocations at EPE.

2. In the second part of the report the advantages of a technique of counter are considered impulses TCI current for research EPE. The availability of a polar part is shown EPE at single change of a direction of a current in metal crystals. The experimental data till EPE with use TCM are adduced. Is analysed Influence of the form of impulses of a current, active width of an amplitude spectrum and role Various parts of a impulse of a current in a realization EPE, and also thermal operation Current at a various porosity of impulses. The brief description of generators is given Current used in TCI the reasons of strengthening EPE at and are indicated Use TCI. B an inference is analyzed an applicability of the theory of electron Wind to explanation EPE in TCI. The bibliography 12 names, number of Figures 8.

Russia, Moscow, 117279, Street of the General Antonova, 4-2-152

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The professor

The doctor of engineering science O.A.Troitsky.

